5.1 Microprobe Analysis

A cross section of a cutting tool from each of the five suppliers was prepared for microprobe analysis, which was considered a more accurate technique than EDS analysis. Those samples were all unetched.

The analyses were done in a Jeol 733 Superprobe at 10 kV accelerating voltage and 40 nA beam current. Two areas of the coatings from the inserts from each supplier were chosen and one line scan in each area was done. The line scans across the coatings of the cutting tool were carried out with a fixed step size of 1 μm between the points. The line scans were not always perpendicular to the coating layers, i.e. the length of the line scan does not always correspond to the thickness of the coating.

5.1.1 Cutting Tool Inserts from Supplier 1

Figures 5.27 and 5.28 show the backscattered electron images of the two selected areas of the coating from an insert from supplier 1 and the line indicates the scan done across the coating.

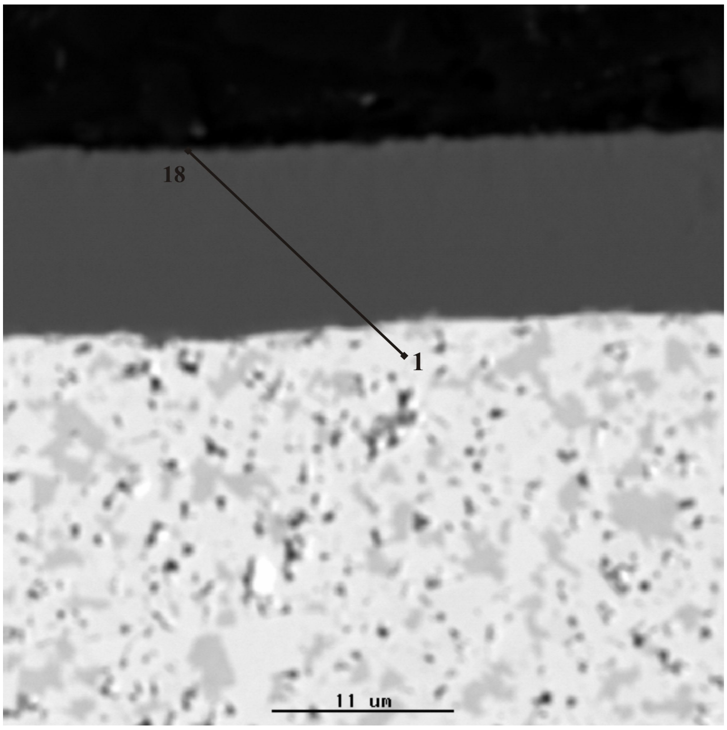

Fig. 5.27: Backscattered electron (BSE) image of Scan 1 of the coatings from an insert from supplier 1 with indicated line scan. The numbers correspond to the points measured across the coating.

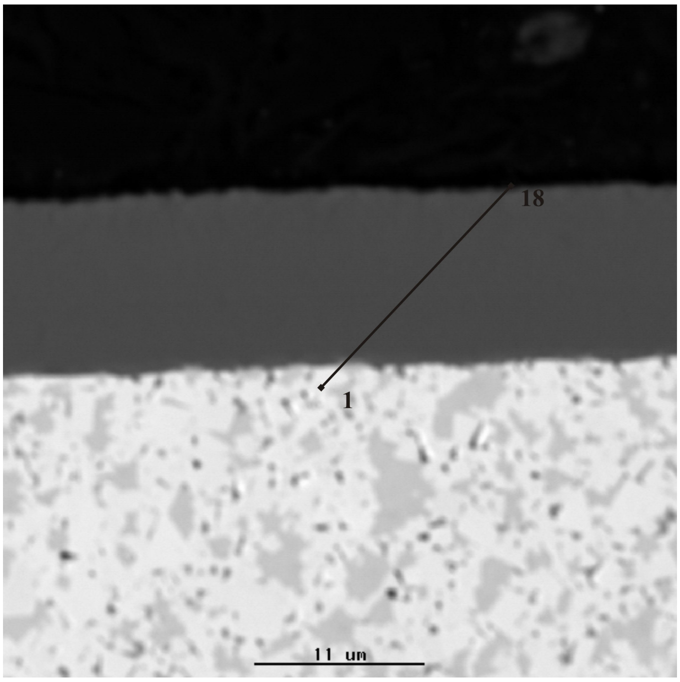

Fig. 5.28: Backscattered electron (BSE) image of Scan 2 of the coatings from an insert from supplier 1 with indicated line scan. The numbers correspond to the points measured across the coating.

The composition of the layers obtained from the line scans in both areas of the coatings from the inserts from supplier 1 in wt.% and at.% can be found in Appendix C.

In each area the line scans started from the substrate and went across the cutting tool coating up to the surface of the coating. Some of the points analyzed were too close to interfaces between different layers. The corresponding results tended to have sums of weight fractions significantly higher than 100 wt.%. As X-ray intensities are absolute measurements against the reference material, matrix effects, such as absorption, have to be taken into account when the measured X-ray intensities (the raw data) are converted to mass fractions of elements. This is done by the so-called “matrix correction” [Scott, 1995]. Those weight fractions much higher than 100 % were discarded because the data reduction during microanalysis assumes that each analysis refers to a homogeneous volume element in the sample (volume size = roughly 1 μm^3). The same data processing applied for the scans in the substrates. In some cases, the sums of weight fractions in the substrates were lower than 100 wt.%. This was due to elements that were present in the substrates, but had been excluded from the analysis (e.g. Co, Nb, Ta).

The line scan data taken along a line which was not perpendicular to the coating layers were converted into measurements in a direction perpendicular to the coating system to relate the composition to the thicknesses of the single coating layers.

The data were converted into graphs “Element wt.% (and at.%) versus distance” (see Figs. 5.29 to 5.32).

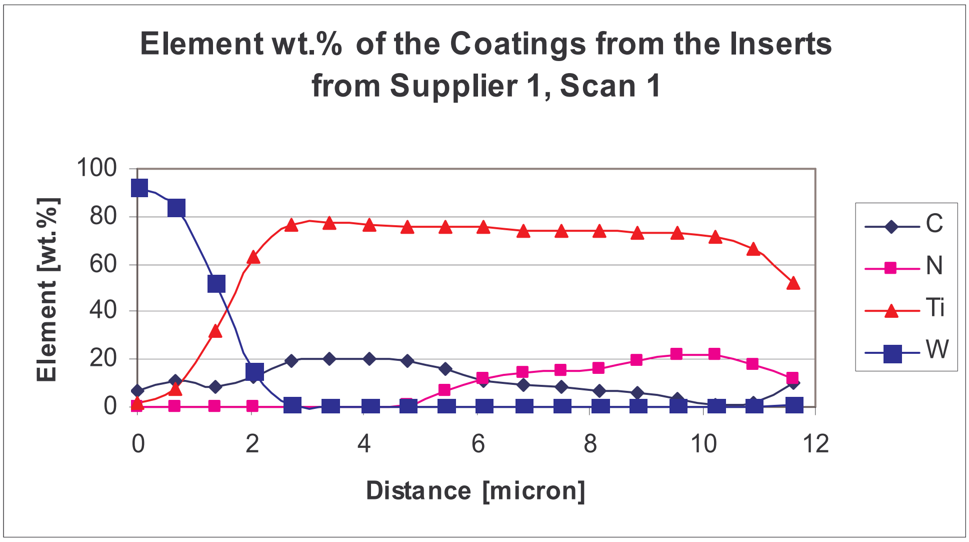

Fig. 5.29: Element wt.% of Scan 1 of the coatings from the inserts from supplier 1

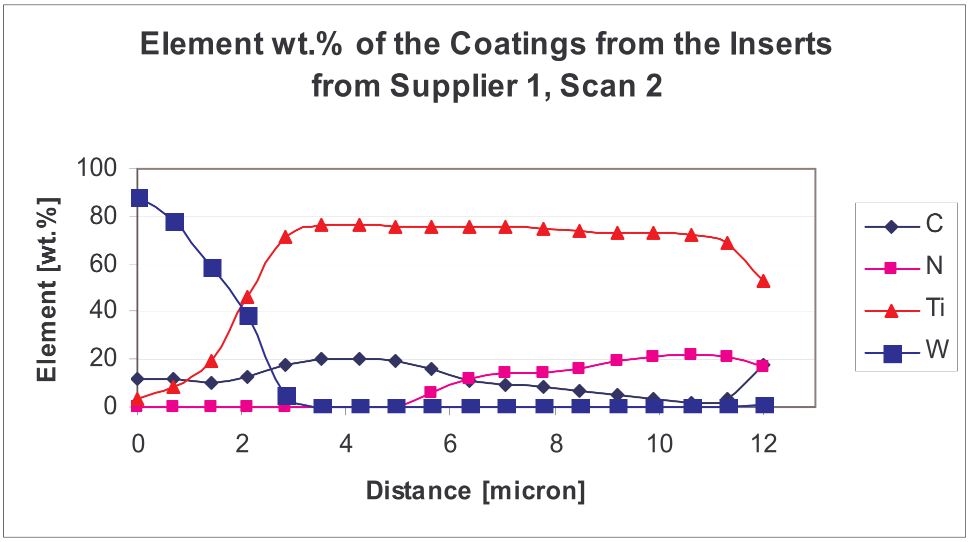

Fig. 5.30: Element wt.% of Scan 2 of the coatings from the inserts from supplier 1

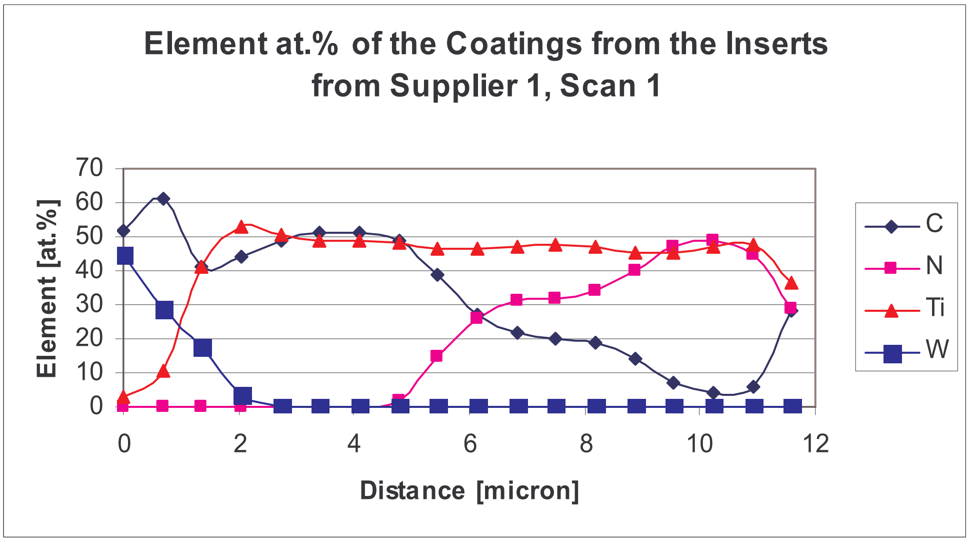

Fig. 5.31: Element at.% of Scan 1 of the coatings from the inserts from supplier 1

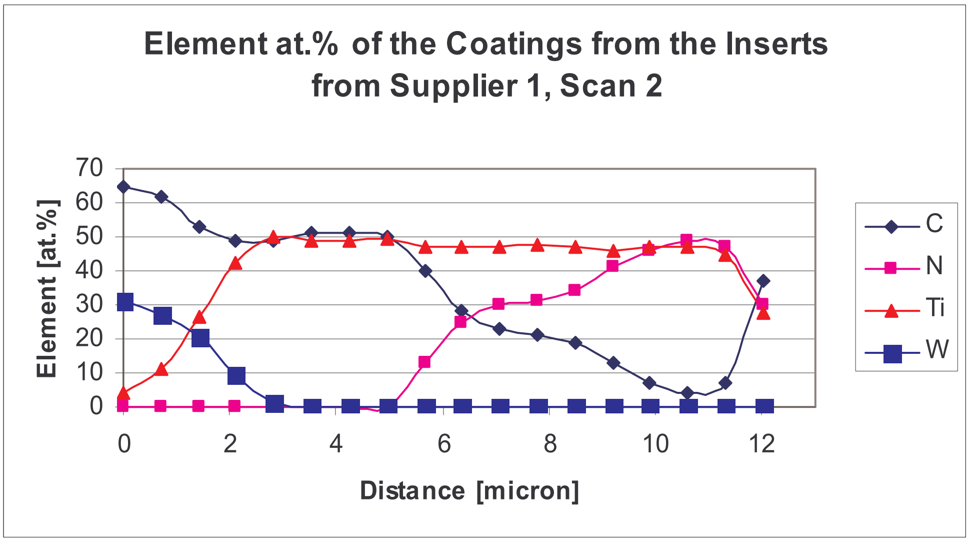

Fig. 5.32: Element at.% of Scan 2 of the coatings from the inserts from supplier 1

The Scan 1 results show that the scan was started from 1 μm below the substrate/coating interface and that the first coating layer (TiC) was approximately 5 μm thick and the second layer (TiCN) approximately 6 μm thick.

The Scan 2 results show that the scan was started from ~ 1.5 μm below the substrate/coating interface and that the first coating layer (TiC) was approximately 5 μm thick and the second layer (TiCN) approximately 7 μm thick.

The results from both scans showed good agreement and good agreement with the EDS results in Table 5.15, as summarized in Table 5.21.

Using the graphs of the at.% measurements in Scans 1 and 2, empirical formulae for each coating layer were obtained (see Table 5.16).

Table 5.16: Empirical formulae for coating layers of the coatings from the inserts from supplier 1

Layer	**Detected elements**	**Empirical formulae**
Substrate		
Layer 1	Ti, C	TiC
Layer 2	Ti, C, N	$Ti_{0.45}C_{0.15}N_{0.40}$

There is good agreement with the empirical formulae obtained by EDS (Table 5.15).

5.1.2 Cutting Tool Inserts from Supplier 2

Figures 5.33 and 5.34 show the backscattered electron images of the two selected areas of the coating from an insert from supplier 2 and the line indicates the scan done across the coating.

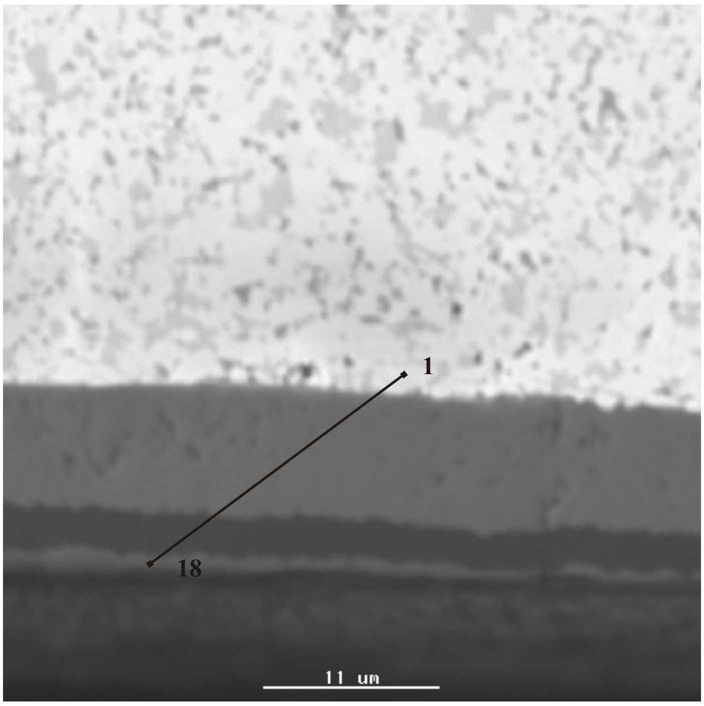

Fig. 5.33: Backscattered electron (BSE) image of Scan 1 of the coatings from an insert from supplier 2 with indicated line scan. The numbers correspond to the points measured across the coating.

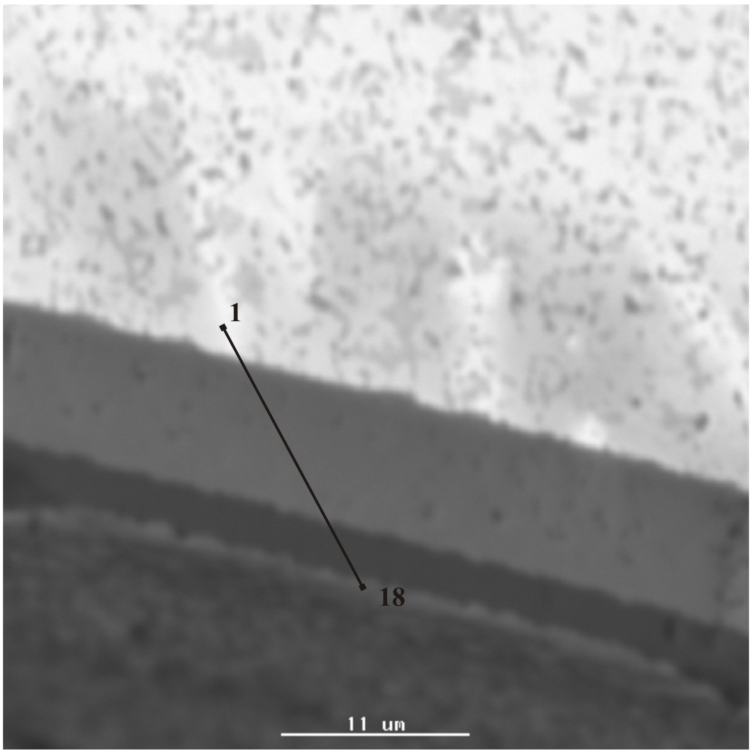

Fig. 5.34: Backscattered electron (BSE) image of Scan 2 of the coatings from an insert from supplier 2 with indicated line scan. The numbers correspond to the points measured across the coating.

The composition of the layers obtained from the line scans in both areas of the coatings from the inserts from supplier 2 in wt.% and at.% can be found in Appendix C.

The outer layer of the coating seemed to be too thin for unambiguous results. That layer might also be rounded due to polishing. The chemical compositions of the last points of the line scans were therefore less reliable than the chemical composition of the other points.

The line scan data taken along a line which was not perpendicular to the coating layers were converted into measurements in a direction perpendicular to the coating system to relate the composition to the thicknesses of the single coating layers.

The data were converted into graphs “Element wt.% (and at.%) versus distance” (see Figs. 5.35 to 5.38).

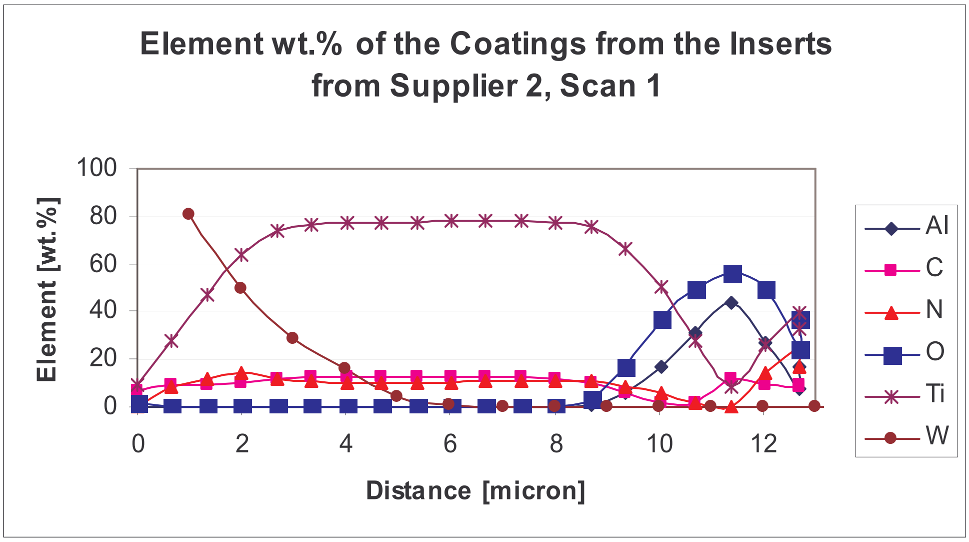

Fig. 5.35: Element wt.% of Scan 1 of the coatings from the inserts from supplier 2

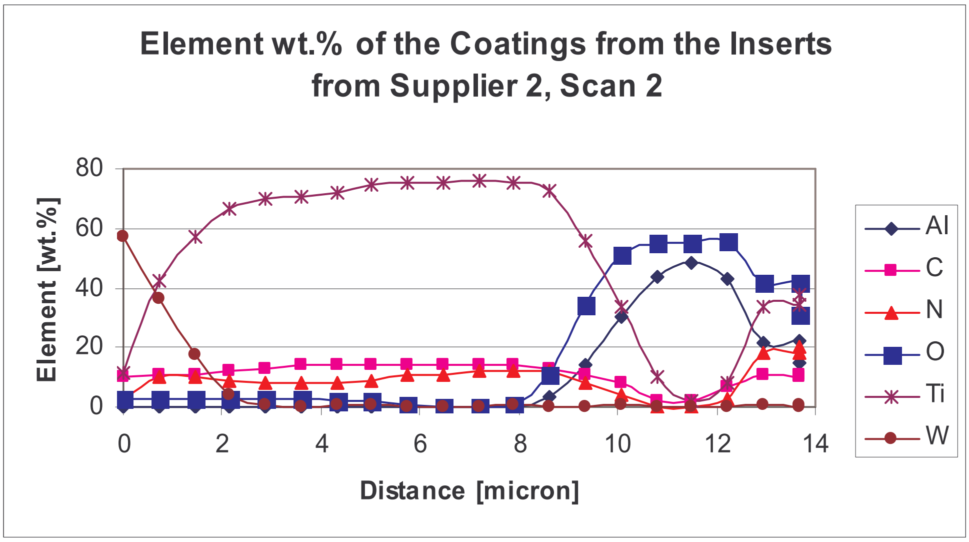

Fig. 5.36: Element wt.% of Scan 2 of the coatings from the inserts from supplier 2

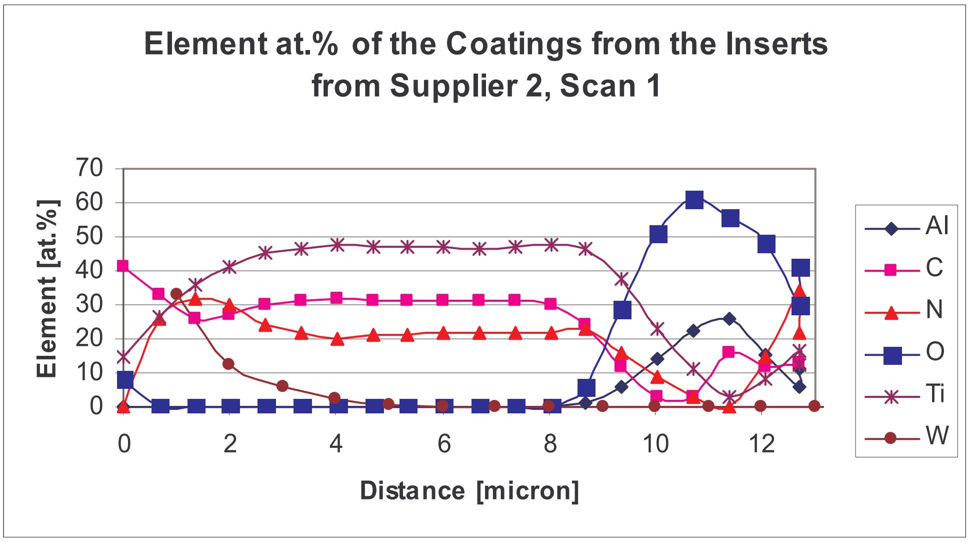

Fig. 5.37: Element at.% of Scan 1 of the coatings from the inserts from supplier 2

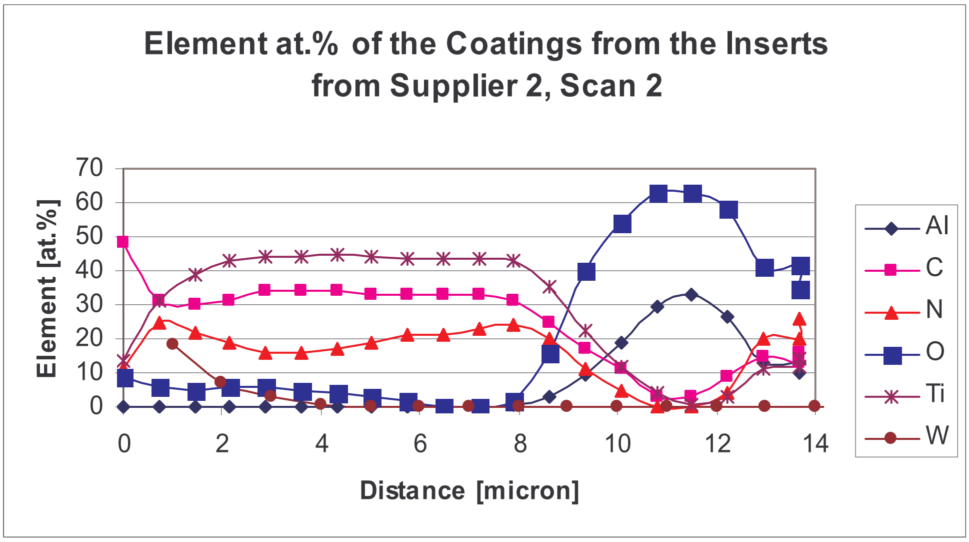

Fig. 5.38: Element at.% of Scan 2 of the coatings from the inserts from supplier 2

The Scan 1 results show that the scan was started from 2 µm below the substrate/coating interface and that the first coating layer (TiCN) was approximately 0.5 µm thin, the second layer (TiCN) was approximately 8 µm thin, the third coating layer (Al_2O_3) was approximately 3 µm thin and the last layer (TiCN) was approximately 0.5 µm thin.

The Scan 2 results show that the scan was started from 2 μm below the substrate/coating interface and that the first coating layer (TiCN) was approximately 1 μm thin, the second layer (TiCN) was approximately 6.5 μm thin, the third coating layer (Al_2O_3) was approximately 3.5 μm thin and the last layer (TiCN) was approximately 1 μm thin.

Using the graphs of the at.% measurements in Scans 1 and 2, empirical formulae for each coating layer were obtained (see Table 5.17).

Table 5.17: Empirical formulae for coating layers of the coatings from the inserts from supplier 2

Layer	**Detected elements**	**Empirical formulae**
Substrate		
Layer 1	Ti, C, N	$Ti_{0.40}C_{0.25}N_{0.35}$
Layer 2	Ti, C, N	$Ti_{0.45}C_{0.35}N_{0.20}$
Layer 3	Al, O	Al_2O_3
Layer 4	Ti, C, N	*

* The element at.% of Ti, C and N of Layer 4 of the coatings from the inserts from supplier 2 did not sum up to 100 %, therefore no empirical formulae could be derived. That layer was thin enough for the beam signal to also detect elements from the neighboring layer.

5.1.3 Cutting Tool Inserts from Supplier 3

Figures 5.39 and 5.40 show the backscattered electron images of the two selected areas of the coating from an insert from supplier 3 and the line indicates the scan done across the coating.

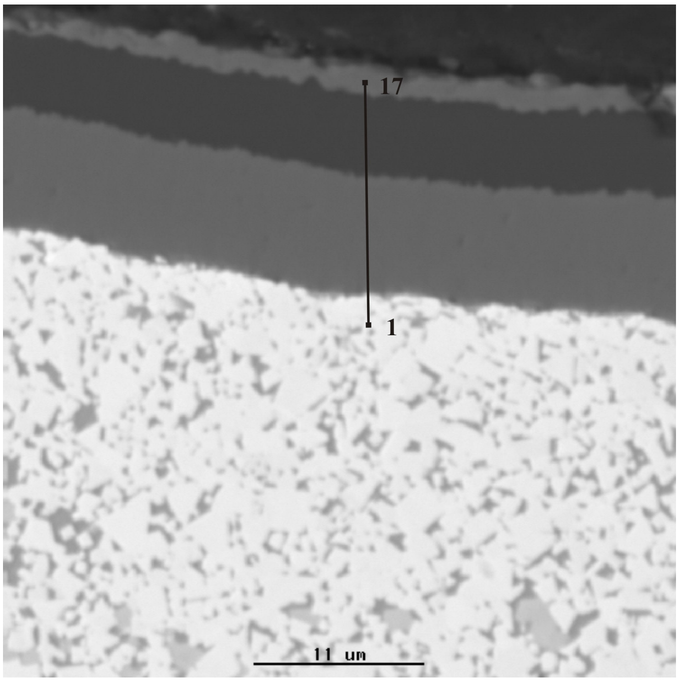

Fig. 5.39: Backscattered electron (BSE) image of Scan 1 of the coatings from an insert from supplier 3 with indicated line scan. The numbers correspond to the points measured across the coating.

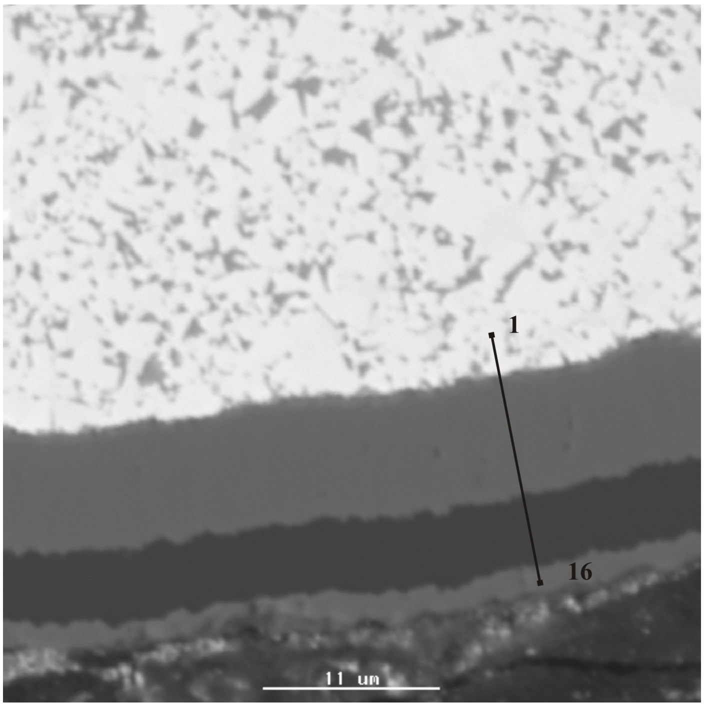

Fig. 5.40: Backscattered electron (BSE) image of Scan 2 of the coating from an insert from supplier 3 with indicated line scan. The numbers correspond to the points measured across the coating.

The composition of the layers obtained from the line scans in both areas of the coatings from the inserts from supplier 3 in wt.% and at.% can be found in Appendix C.

The data were converted into graphs "Element wt.% (and at.%) versus distance" (see Figs. 5.41 to 5.44).

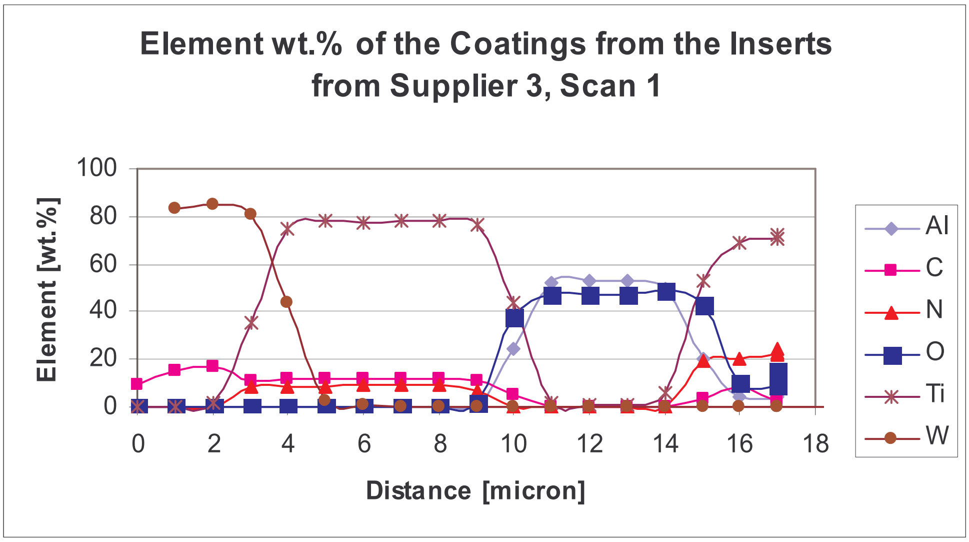

Fig. 5.41: Element wt.% of Scan 1 of the coatings from the inserts from supplier 3

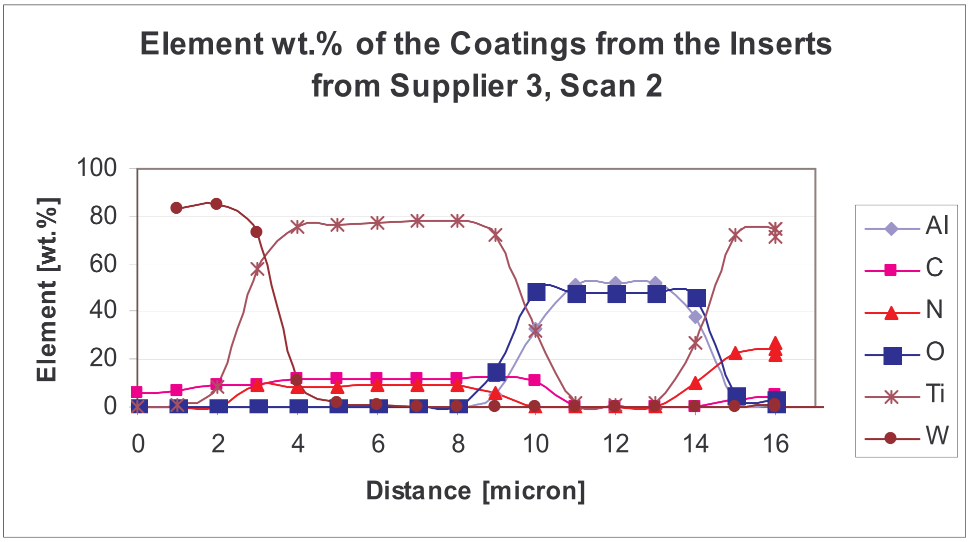

Fig. 5.42: Element wt.% of Scan 2 of the coatings from the inserts from supplier 3

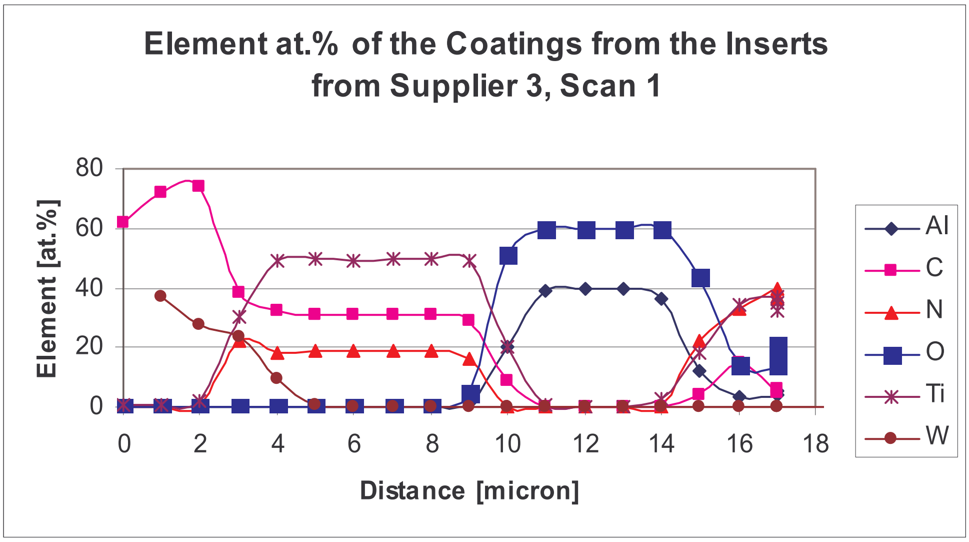

Fig. 5.43: Element at.% of Scan 1 of the coatings from the inserts from supplier 3

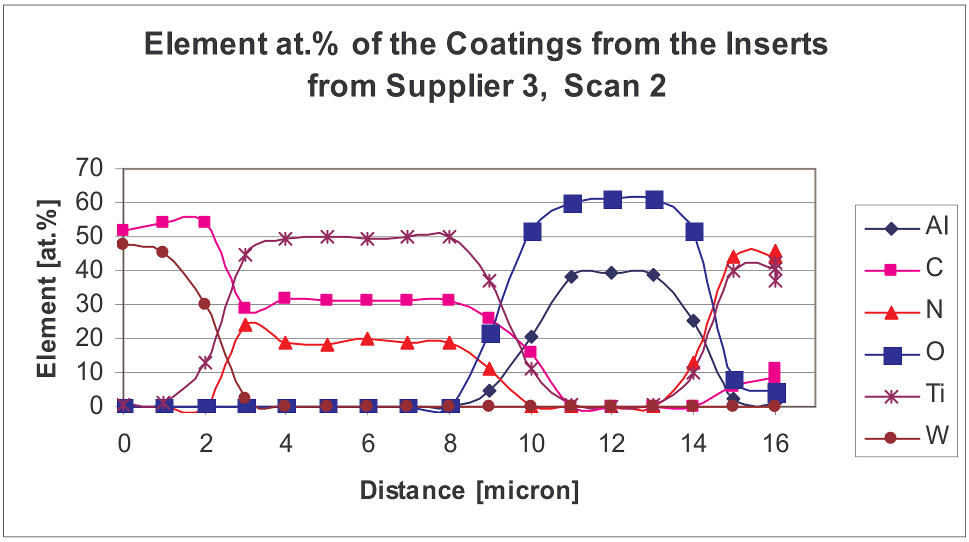

Fig. 5.44: Element at.% of Scan 2 of the coatings from the inserts from supplier 3

The Scan 1 results show that the scan was started from 3 μm below the substrate/coating interface and that the first coating layer (TiCN) was approximately 1 μm thin, the second layer (TiCN) was approximately 6 μm thin, the third coating layer (Al_2O_3) was approximately 5 μm thin and the last layer (TiCN) was approximately 2 μm thin.

The Scan 2 results show that the scan was started from 2 μm below the substrate/coating interface and that the first coating layer (TiCN) was approximately 1 μm thin, the second layer (TiCN) was approximately 6 μm thin, the third coating layer (Al_2O_3) was approximately 5 μm thin and the last layer (TiCN) was approximately 2 μm thin.

The results from both scans showed good agreement and good agreement with the EDS results in Table 5.15, as summarized in Table 5.21.

Using the graphs of the at.% measurements in Scans 1 and 2, empirical formulae for each coating layer were obtained (see Table 5.18).

Table 5.18: Empirical formulae for coating layers of the coatings from the inserts from supplier 3

Layer	**Detected elements**	**Empirical formulae**
Substrate		
Layer 1	Ti, C, N	$Ti_{0.45}C_{0.30}N_{0.25}$
Layer 2	Ti, C, N	$Ti_{0.50}C_{0.35}N_{0.15}$
Layer 3	Al, O	Al_2O_3
Layer 4	Ti, C, N	$Ti_{0.45}C_{0.10}N_{0.45}$

There is good agreement with the empirical formulae obtained by EDS (Table 5.15).

5.1.4 Cutting Tool Inserts from Supplier 4

Figures 5.45 and 5.46 show the backscattered electron images of the two selected areas of the coating from an insert from supplier 4 and the line indicates the scan done across the coating.

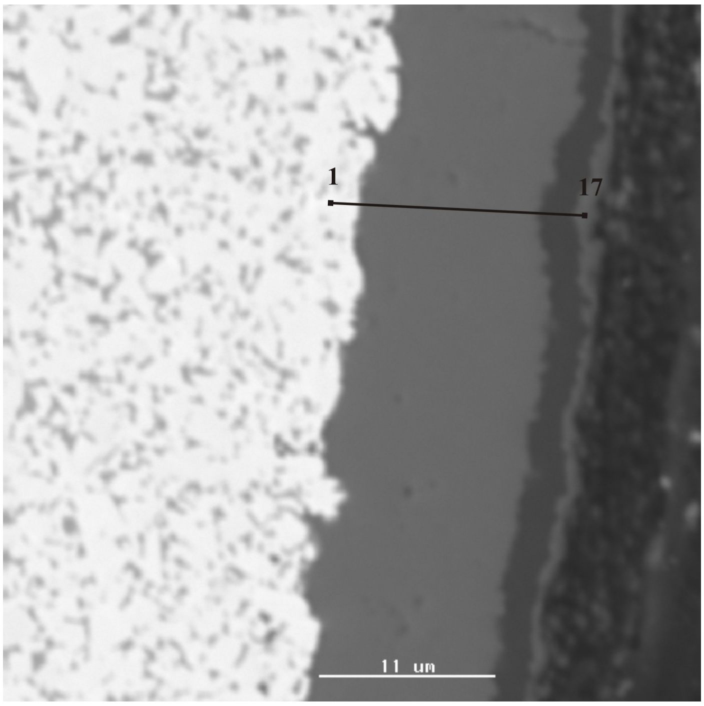

Fig. 5.45: Backscattered electron (BSE) image of Scan 1 of the coatings from an insert from supplier 4 with indicated line scan. The numbers correspond to the points measured across the coating.

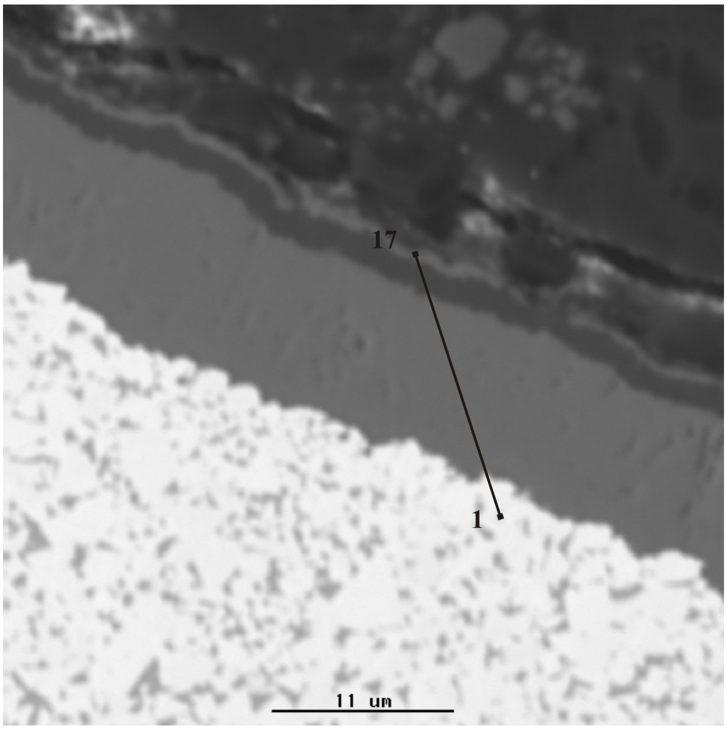

Fig. 5.46: Backscattered electron (BSE) image of Scan 2 of the coatings from an insert from supplier 4 with indicated line scan. The numbers correspond to the points measured across the coating.

The composition of the layers obtained from the line scans in both areas of the coatings from the inserts from supplier 4 in wt.% and at.% can be found in Appendix C.

The line scan data taken along a line which was not perpendicular to the coating layers were converted into measurements in a direction perpendicular to the coating system to relate the composition to the thicknesses of the single coating layers.

The data were converted into graphs "Element wt.% (and at.%) versus distance" (see Figs. 5.47 to 5.50).

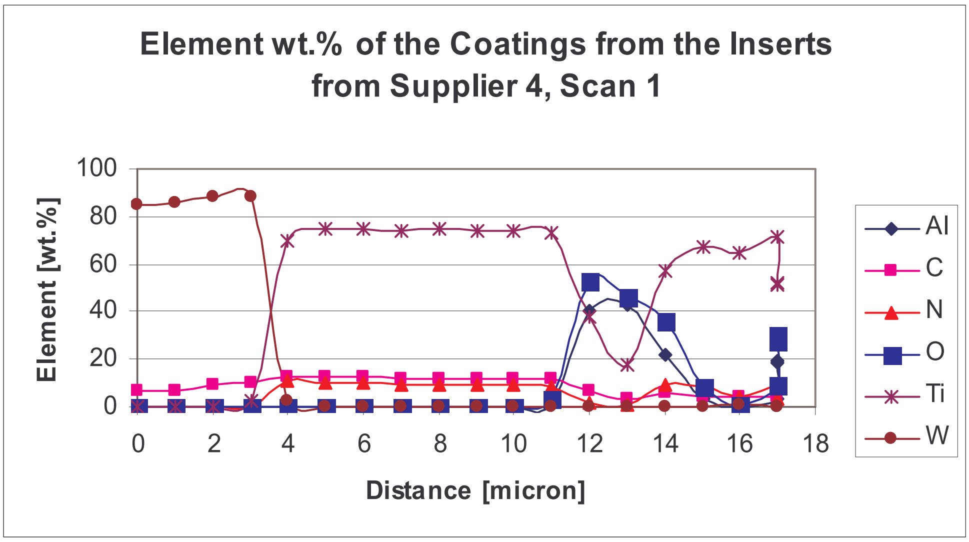

Fig. 5.47: Element wt.% of Scan 1 of the coatings from the inserts from supplier 4

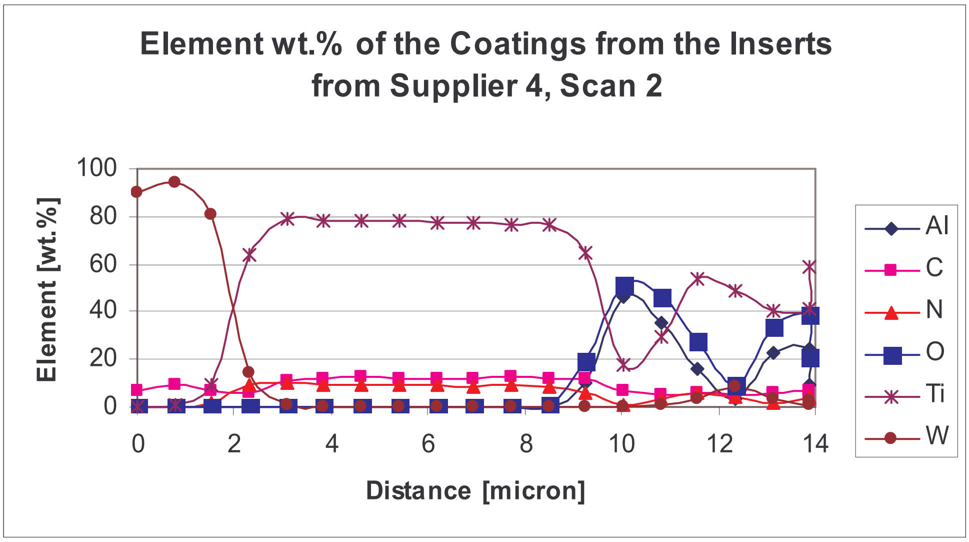

Fig. 5.48: Element wt.% of Scan 2 of the coatings from the inserts from supplier 4

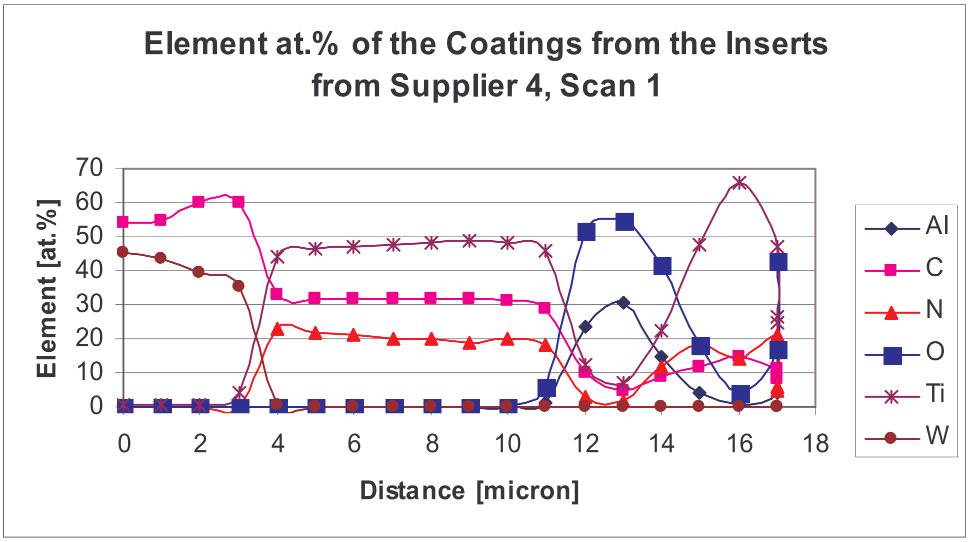

Fig. 5.49: Element at.% of Scan 1 of the coatings from the inserts from supplier 4